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ON FILTER SELF-ABSORPTION AND LOADING CHARACTERISTICS

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ON FILTER SELF-ABSORPTION AND LOADING CHARACTERISTICS

by

M. Grenier*

ABSTRACT

A fiber filter and a membrane type filter were examined to measure the effect of aerosol type and size, sampling flow rate and inert dust loading on self-absorption of alpha radiation. Air sampling of alpha radiation was conducted (using both types of filters) in relatively dust free environments (laboratory test facilities with aerosol concentrations as low as 1.2×10^3 cm^{-3}) as well as very dusty environments (underground uranium mines). The total self-absorption of alpha activity varied between 2% and 8% for the fiber filter used (fiberglass type) and was found to be negligible for the membrane filter. Results show that the self-absorption of alpha activity in fiber filters depends on the concentration and size of aerosol sampled as well as on the sampling flow rate used. This is in agreement with the theories of particulate penetration through fiber filter media.

The effect of filter dust loading on instruments operating on alpha spectroscopic principles was determined. Problems arising from dust loading such as spectral peak shifting and peak broadening can lead to gross errors in measurements. Experimental results show that these effects can be reduced in continuous sampling instruments by reducing the flow rate and by the use of membrane filters which have lower self-absorption characteristics.

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DÉTERMINATION DES CARACTÉRISTIQUES D'AUTO-ABSORPTION ET DE CHARGEMENT DES FILTRES

par

M. Grenier*

RÉSUMÉ

On a examiné un filtre en fibre ainsi qu'un filtre à membrane dans le but de mesuré l'effet du genre et de la dimension des aérosols, du débit de prélèvement et du chargement de poussières inertes sur l'auto-absorption du rayonnement alpha. Le prélèvement de rayonnement alpha dans l'air a été effectué (à l'aide des deux genres de filtre) dans un environnement relativement exempt de poussière (les installataions d'essai en laboratoire avaient des concentrations d'aérosols aussi faibles que 1.2×10^3 cm^3) ainsi que dans un environnement très poussiéreux (mines souterraines d'uranium). L'auto-absorption totale de l'activité alpha variait entre 2 et 8 % avec l'utilisation du filtre en fibre (fibre de verre) et s'avérait négligible pour ce qui est du filtre à membrane. Les résultats ont démontré que l'auto-absorption de l'activité alpha sur les filtres en fibre dépendait aussi bien de la concentration et de la dimension des aérosols prélevés que du débit de prélèvement utilisé. Ceci concorde avec les théories de la pénétration des particules à travers la matière fibreuse du filtre.

On a de plus déterminé l'effet que le chargement des filtres avec de la poussière avait sur les appareils fonctionnant selon les principes spectroscopiques alpha. Les problèmes soulevés par le chargement de poussières, tels le déplacement du pic spectral et l'élargissement du pic peuvent occasionner de sérieuses erreurs dans les mesures. Les résultats d'essai indiquent que ces effets peuvent être réduits dans les instruments à prélèvement continu par la diminution du débit et l'utilisation de filtres à membrane possédant des caractéristiques d'auto-absorption moins élevées.

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INTRODUCTION

The optimizing of sampling procedures and the need to minimize errors both while sampling and analyzing environmental radioactive contaminants remain a very contemporary concern. The possible sources of error may be classified as indeterminate, such as the uncertainty brought about by the random nature of radioactive decay, or as determinate, such as instrument malfunction, or questionable experimental procedures. Errors of the first type are beyond control, whereas errors of the determinate, or systematic type, may be eliminated or kept to an acceptable minimum (1). While most sources of determinate error are easily dealt with, some such as self-absorption and the effect of filter loading are more of a problem and need to be well understood in order to better deal with them.

In the case of low energy radiation, self-absorption is loosely defined as total or partial absorption of ionizing radiation energy in the whole sample matrix. For the purpose of discussion, it is useful to differentiate between self-absorption in the collected dust load, SA_D, and self-absorption in the filter material itself, SA_F. When measuring self-absorption in the field, however, the total self-absorption is measured without distinction between SA_D or SA_F. Self-absorption will cause experimentally observed counting rates to be as much as 10% lower than the actual disintegration rate of the sample. Self-absorption is brought about partly by collection in depth of particulates within filter material. This is especially true of so-called depth or fiber filters. Factors such as sampling flow rate, or filter face velocity, size distribution of aerosol sampled, and dust load will be shown to have an effect on the degree of self-absorption.

Filter loading, is a factor that affects self-absorption as defined above. More importantly, however, filter loading and partial self-absorption

in dust and filter material is a major cause of peak, or energy shifting, and peak broadening when alpha-spectroscopy is being performed on samples. Many state-of-the-art environmental radiation monitors operate in a continuous fashion for extended periods of time. In some cases these instruments operate on alpha-spectroscopic principles and dust loading can lead to erroneous readings.

In this report, the theory of filtration will be reviewed briefly. The phenomena that affect self-absorption will be studied experimentally for glass fiber filters and the results will be compared to theoretical predictions. The effects of filter loading will be demonstrated for both glass fiber and membrane filters. Recommendations will be made on how to reduce the effects of these problems as they apply to actual sampling situations.

THEORY

GLASS FIBER FILTERS

Fiber filters are often called depth filters since they rely on the depth of the filter mat to retain particles (2). These filters usually consist of small randomly distributed fibers that are supported by larger fibers that act as a skeleton. In this type of filter, the inter-fiber distance is large compared to particle radius, and for this reason various phenomena are competing and contribute to particle trapping. Figure 1 illustrates some of these phenomena (3). In every case it is assumed that once the particles come into contact with the fiber they remain attached to it. In the size range of interest, the bonds created by surface tension are relatively strong so that this type of assumption is reasonable.

Figure 1c illustrates trapping by interception. In this case particles come in contact with the fiber without deviating from the streamlines and so this means of trapping particles is basically independent of air stream

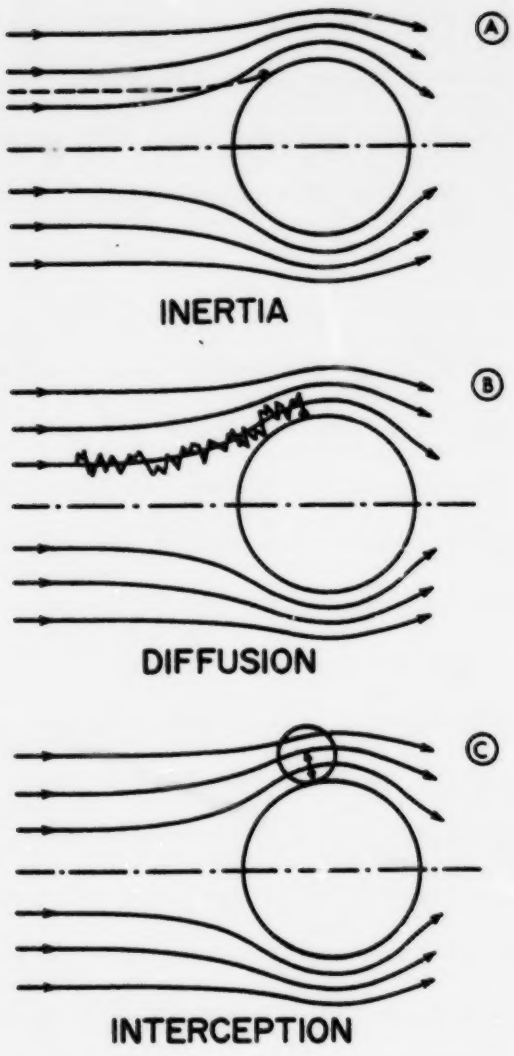


Fig. 1 - Schematic representation of particle capture in fiber filters by inertial effects, by diffusion and by interception. Note: the large circle represents a cross-sectional view of the fiber.

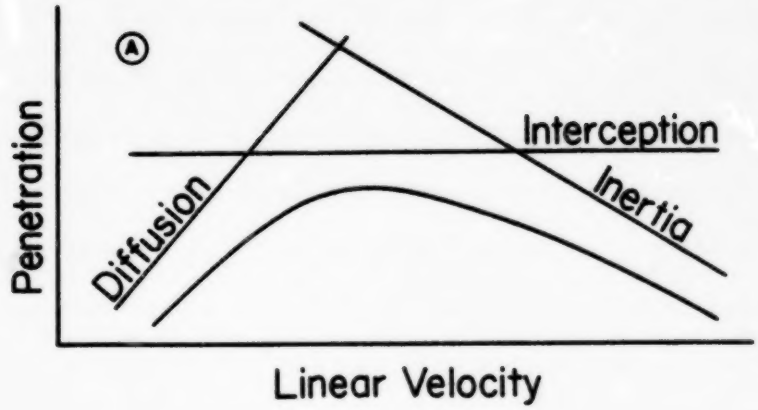
velocity. Particle radius on the other hand is an important variable and capture by interception is only significant for large size particles.

Figure 1a shows the effect of inertial forces. This happens when particles do not follow the streamlines mentioned earlier. Inertial forces cause these particles to collide with the fiber directly and to become attached. From the definition of inertia it can be inferred that heavier particles as well as higher air stream velocities will enhance capture by inertial effects.

Figure 1b shows capture by diffusion. Random collisions with gas molecules in air cause what is commonly called Brownian motion. While travelling in the general direction set by the streamlines, particles follow a very random path that can cause them to come in contact with filter fibers and be collected. This capture method is enhanced for smaller size particles and lower airstream velocities. When particles become larger, inertia and interception are more important trapping factors.

The effect of airstream or linear face velocity is shown in Figure 2a (3) and it may be summarized as follows. At low velocities diffusion is a large factor for particle removal and penetration into filter material is small. As velocity increases the probability of attachment to fibers by diffusion is reduced which increases penetration. An increased air velocity on the other hand causes particles to be trapped by inertia so that penetration is eventually reduced as air velocity is further increased. The effects of interception are not assumed to be affected by air velocity. All of the above discussion assumes a constant particle size distribution throughout. Other mechanisms for capture include those that might be enhanced by particle charge, especially in the .1 to 1 μm range (3), and also gravitational settling for very massive particles.

Another factor affecting penetration into filter media is particle size



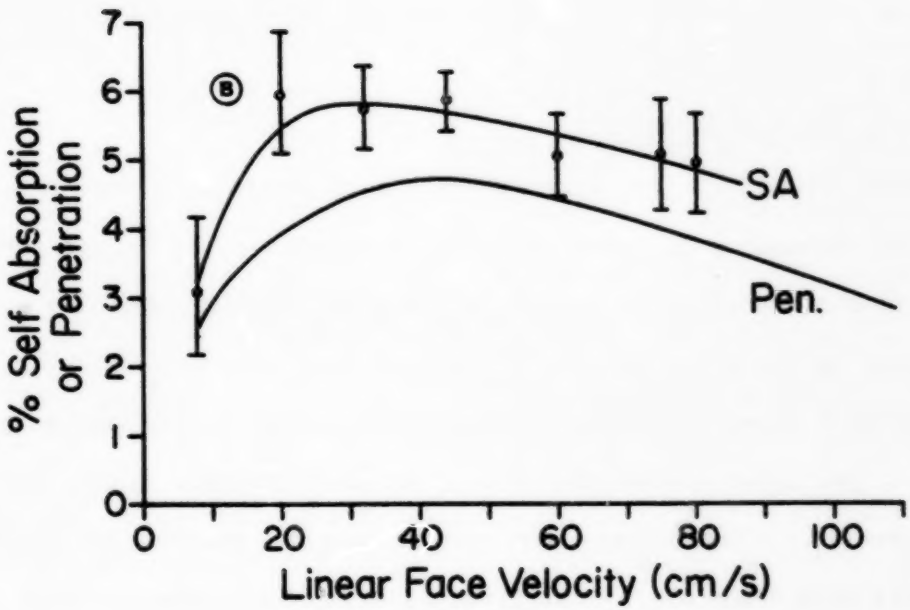


Fig. 2a - The effect of varying the flow rate (linear velocity) on aerosol penetration in fiber filters.

Fig. 2b - The effect of flow rate on aerosol penetration for fiber filters (bottom curve) compared to the effect of flow rate on radioactive aerosol self-absorption as measured experimentally for glass fiber filters.

distribution. The major mechanism affecting small particles is diffusion while larger particles are trapped, mostly by inertial forces. This implies that there should be peak penetration for an intermediate particle size. This size is thought to be situated between .1 and .3 μm in most cases when fiber filters are concerned. This fact is of considerable importance since it is known that most environmental radioactive contaminants are found to be attached preferentially to particles in the .1 to .2 μm size range (4).

Dust load is yet another factor affecting the penetration of particles in filter media. As radioactive aerosols and inert dusts collect onto filter fibers, the interstitial space between fibers diminishes. This enhances every one of the capture mechanisms described earlier. At first glance this might seem to be beneficial, but several drawbacks exist, one of which is filter clogging.

PENETRATION AND SELF-ABSORPTION

Penetration is a phenomenon whereby dust particles manage to elude the collection mechanisms described earlier and penetrate the filter completely. Partial penetration could be defined as the collection of particles at varying depths into filter media because of the intrinsic structure of depth filters. This in itself is not a problem as long as fiber filters are used as part of a filter, or prefilter system aimed solely at dust removal. In some practical cases, however, fiber filters are used to sample radioactive contaminants. This might seem to be unwise but fiber filters have the advantage of being able to accept a much larger dust load than membrane filters in situations where dust concentration is high, or sampling periods are long. Hence, partial penetration becomes a definite problem in sampling procedures when fiber filters are involved, since it invariably leads to alpha and beta particle self-absorption. Depending on the filter material, the flow rate used and aerosol size, self-absorption may vary between 1 to 10%, and beyond.

It seems then that penetration and self-absorption should be very closely related. The physical princiles governing fiber filter penetration have been studied at length (3). The present work will attempt to show that the principles governing partial penetration (collection at depth) also govern radiation self-absorption.

MEMBRANE FILTERS

These filters are commonly used for environmental sampling. They are made of such materials as cellulose triacetate, PVC and mixed cellulose esters (2). These materials are bound together by intermolecular forces, and therefore, contain no glues or other binders. These filters contain no fibers and have a void volume of about 80% (5). Pore sizes are fairly uniform for a given type of filter and may vary approximately between .1 and 1.2 μ for different types of filters. The thickness of the membrane varies from 100 to over 200 μm.

The self-absorption of alpha or beta activity due to filter medium for membrane filters is usually considered to be negligible since a large portion of the filter media is void volume and particles are generally thought to be collected at very shallow depths. In fact partial penetration, and hence the self-absorption may be kept consistently as low as, or lower than 2%, if careful experimental procedures are followed. The most serious problem associated with membrane filters is that for applications where extended sampling periods are required, dust loading will become a problem that will inevitably lead to pump failure. Dust loading also leads to self-absorption in the dust sample which in turn causes total and partial alpha-energy absorption. This creates serious problems when filters are being analyzed by instruments that cannot correct for alpha peak shifting and broadening when alpha-spectroscopic analysis is used. These problems will be demonstrated experimentally further on and possible solutions will be suggested.

INSTRUMENTS AND METHODS

Two types of filters were used, one of the depth type (Gelman Glass Fiber Filters Type A/E) and one membrane filter (Millipore .8 μm pore size Type AA). The filters used were always 25 mm in diameter and appropriate Delrin Open Filter Holders were used. Samples were taken at various flow rates using a Sierra Instrument Model 113 Constant Flow Air Sampler. This sampler can operate in the 1 to 20 L/min range with little fluctuation for extended periods of time. The counting of filters for activity in order to determine self-absorption was performed using Tri-Met TM-372A Scalers. These scalers consist of a photomultiplier tube that is sensitive to photons emitted by a silver activated ZnS screen that faces the radioactive sample.

The sampling and analysis procedures followed were basically simple and aimed at simulating realistic sampling methods. Filters were carefully placed in the filter holder which was fitted on the pump. Sampling then took place at a predetermined flow rate for a given period of time which varied in order to get a proper level of activity on the filter. After this was done, filters were left aside for a period of at least three hours. The reasons for this are, first, to let all of the radon progeny decay away, and secondly to let the thoron progeny come to equilibrium in order to have the alpha decays from ThC and ThC' proceed with the half-life of ThB (10.6 h). This procedure was followed in order to allow the counting of filters to be performed more accurately without having to worry about errors being introduced because of decay rate changes.

Some samples were taken in an artificial thoron environment within a Radon/Thoron Test Facility (RTTF) where the aerosol concentration was kept at less than 1.0×10^4 particles per cm^3. Some other samples were obtained from an underground uranium mine where both radon and thoron daughters are found

in the atmosphere. Underground samples were taken to demonstrate the effects of loading. In the case of loading experiments, sampling took place over periods of 24 h, or 7 days, at .5 L/min and 7 L/min, respectively, for both membrane and fiber filters.

The formula used to obtain the self-absorption coefficient was obtained from (6):

$$SA = \frac{C_2 - C_3}{2C_1 + C_2 - C_3} \qquad \text{Eq 1}$$

where, SA is self-absorption within the filter material.

C_1 is the count rate obtained from the front of the filter.

C_2 is the count rate obtained from the back or less active side of the filter.

C_3 is the count rate obtained from the front, while another similar filter disk is superimposed.

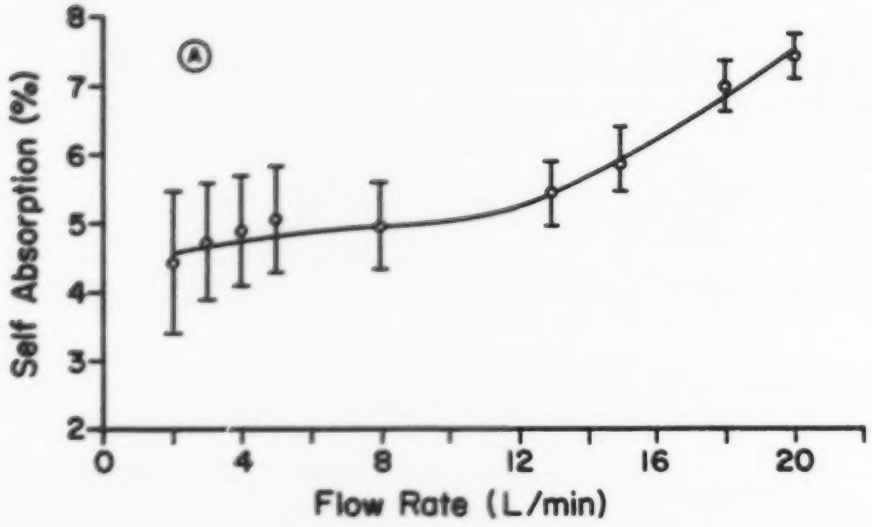
This equation may be derived by following a procedure outlined in (5), the details of which appear in the Appendix.

RESULTS AND DISCUSSION

SELF-ABSORPTION IN FIBER FILTERS

Referring back to the bottom half of Figure 2, the lower curve is a plot of fiber filter penetration versus linear sampling velocity (3). The upper curve (with error bars, displays self-absorption as a function of linear sampling velocity for glass fiber filters. This graph shows that there is indeed a close relationship between self-absorption and fiber filter penetration. From that we can deduce that the physical phenomena responsible for penetration (diffusion, inertia and interception) also have a similar effect on observed self-absorption. The experimental curve is an average over three separate experiments conducted under similar conditions. The error bars

were calculated from mean deviations. The three experiments took place in an environmental chamber where the aerosol concentration was monitored and kept at approximately 5.0 x 10^3 particles/cm^3. The experimental self-absorption curve peaks at about 30 cm/s (approximately 7 L/min for a 25 mm filter). Therefore, one way to minimize self-absorption would be to lower the flow rate in order to take advantage of the diffusion effects at lower linear velocities. Beyond 20 cm/s inertial forces reduce the self-absorption, but the linear velocity would have to be increased to a point where it might become impractical in order to affect the self-absorption appreciably. It should be noted that a different aerosol distribution would cause the peak to shift to the right for smaller aerosol and to the left for larger aerosol. A good example of this is shown experimentally in Figure 3. In the top graph self-absorption is plotted as a function of flow rate for 25 mm glass fiber filters. For the purpose of comparison with Figure 2, 20 L/min corresponds roughly to a linear velocity of 80 cm/s. In the top graph of Figure 3, it can be seen that self-absorption does not peak as it does in Figure 2. This can be explained by examining the bottom graph in Figure 3 which shows an average aerosol concentration of approximately 1,200 particles/cm^3 during the time when self-absorption measurements were taken (between 9 and 11 am). Experimental data reported elsewhere (7), shows that at such low aerosol concentrations, close to 70% of RaA daughters would be unattached to aerosols in a natural environment. Although we are dealing with thoron daughters in this case, it is not hard to imagine that with an aerosol concentration of 1,200 cm^{-3}, a large percentage of radionuclides would be in the unattached form. These free nuclides have small effective radii and hence their diffusion coefficient is in the vicinity of .06 cm^2/s (8). For this reason, it is believed that the main mechanism affecting self-absorption as a function of flow rate in Figure 3 is diffusion. In other words, the peak one would



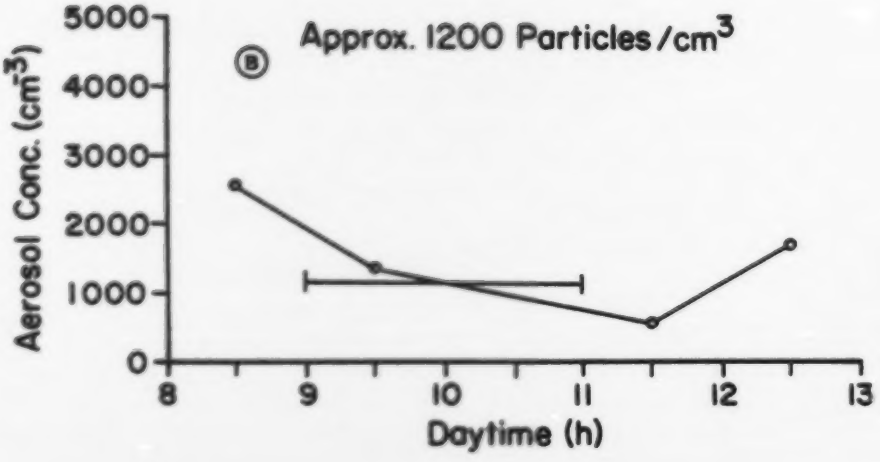


Fig. 3a - Radioactive aerosol self-absorption versus flow rate as calculated for fiber glass filters at the aerosol concentration shown in 3b.

Fig. 3b - Aerosol concentration in calibration facility at the time the above self-absorption measurements were made.

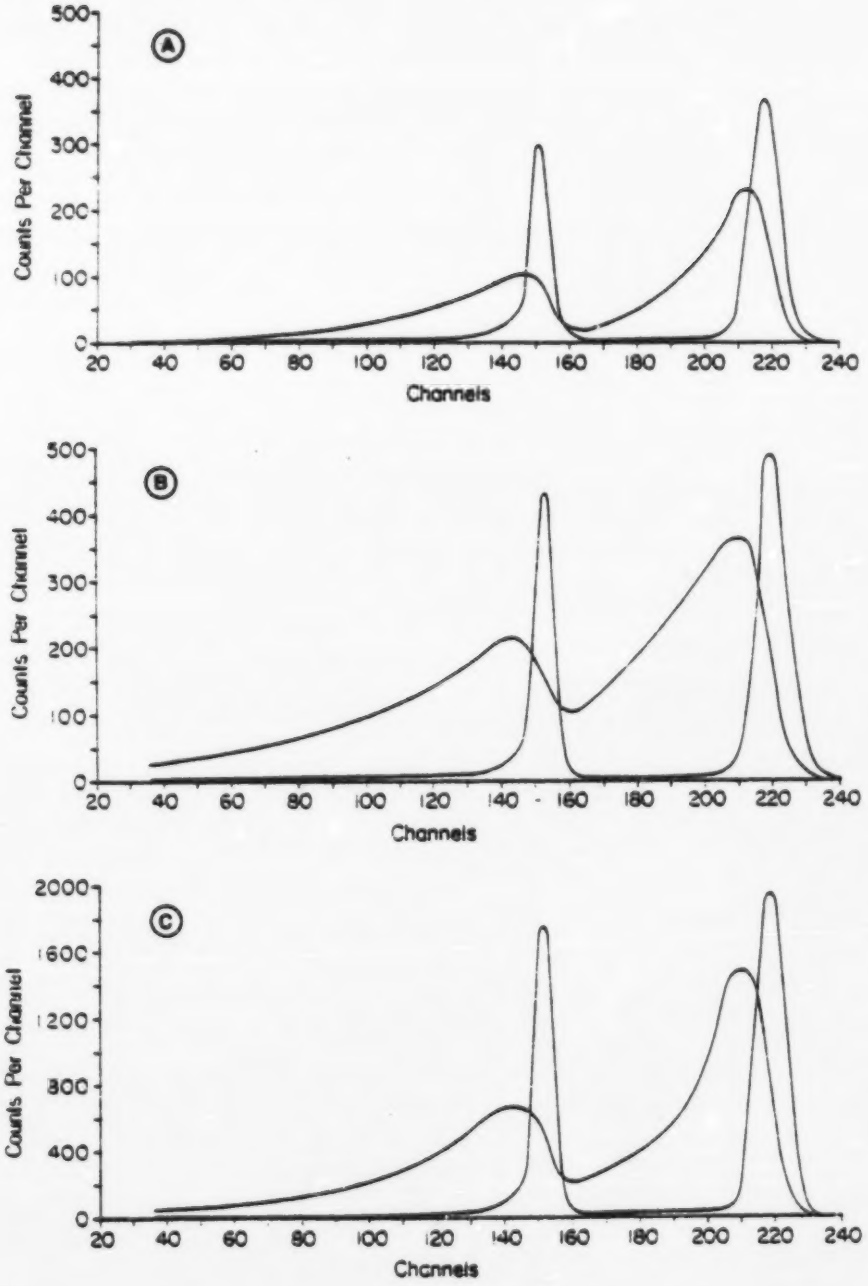


Fig. 4 - Alpha-spectroscopy analysis performed on various radioactive dust samples. Graph A shows the effect of dust load on peak spreading for glass fiber filters at .5 L/min for a 7-day sampling period. Graph B for glass fiber filters at 7 L/min for 24 hours; and graph C for a membrane filter at .5 L/min for 24 hours.
Note: in every case the narrow peak spectrum from a membrane filter whose activity was obtained from the Pylon source described before is included for comparison purposes.

expect from examining Figure 2 has been shifted to the right because the mean radius of radioactive aerosol has been effectively reduced via an increase of the unattached fraction.

FILTER LOADING

Figure 4 shows alpha-spectroscopy analyses performed on various filtered samples. These were obtained by examining the samples using a ruggedized Si-barrier detector within a vacuum chamber. Every graph consists of two superimposed alpha-spectroscopy analyses. The narrow peaked distribution is obtained from a membrane filter that was placed inside a Pylon Ltd. Type 190 thoron daughter source. The activity collected on the surface of the filter is deposited by impaction in a very thin layer. This is done in order to simulate a situation where SA_D and SA_F are very low. This is used for comparison purposes. The broader peaked distributions are from heavy dust load samples collected in underground uranium mines. In both cases, the right peak belongs to the higher energy ThC', while the left peak belongs to ThC.

The graph on Figure 4a shows a sample taken over a period of one week at a flow rate of .5 L/min using a glass fiber filter. Comparison with the Pylon sample shows a very pronounced spread in alpha activity peaks. This spread is mainly caused by partial absorption of alpha-energy within the inert dust build-up on the filter. Figure 4b is a similar sample taken at 7 L/min for a period of about 24 hours. It can be seen that the spread is greater than that of Figure 4a. Intuitively, one might expect the degree of spread to be alike, but this is not so, mainly because in the .5 L/min case, most of the activity that was collected at the beginning of the sampling has decayed away and is not absorbed by the dust collected afterwards. In other words, most of the activity counted on the seventh day comes from very close to the surface of the dust sample. In the case of the 7 L/min sample (24 hours), however, the spread is more pronounced since partial alpha-energy absorption is taking

place at all depths within the sample. From Figures 4a and 4b we can deduce that peak spreading may be minimized if lower flow rates are used. This practice will also reduce pump clogging due to filter loading. One negative aspect of reducing the flow rate is that the collected activity is reduced proportionally, which could affect counting statistics.

Figure 4c shows the result of a 7 L/min sample taken over a period of 24 hours on a membrane filter. Even at 7 L/min it appears that peak spreading is not as severe as for the case of the fiber filter in Figure 4b. This would tend to indicate that some of the energy absorption is taking place within the glass fiber mat itself in Figure 4b. Hence, whenever it is practically possible, a membrane filter should be used. These filters, however, will tend to load more quickly and usually require the use of servo-controlled pumps to adjust for changes in pressure drop across the filter.

Figure 5 shows the effect of filter loading on continuous radiation monitors that use discriminator voltages or energy windows to differentiate between radionuclides on the basis of alpha energy. Both samples were obtained by counting the respective filters using an instrument that operates on the discriminating principles described above. Such an instrument usually generates three voltages that will define energy windows such as the ones shown in Figure 5. The lowest voltage is set in order to eliminate low energy noise that might be found in the low energy region (prior to channel 36, see Figure 5). This voltage and the intermediate one define the region between channels 36 and 106 where RaA (6.0 MeV) and ThC (6.1 MeV) are detected. The intermediate and the high voltages define a region between channels 106 and 135 where RaC' (7.7 MeV) is detected. Finally, the region beyond channel 135 is used to detect ThC' (8.8 MeV).

Figure 5a shows a thoron daughter spectrum from a membrane filter obtained from the Pylon Type 190 source. This graph shows a minimal peak

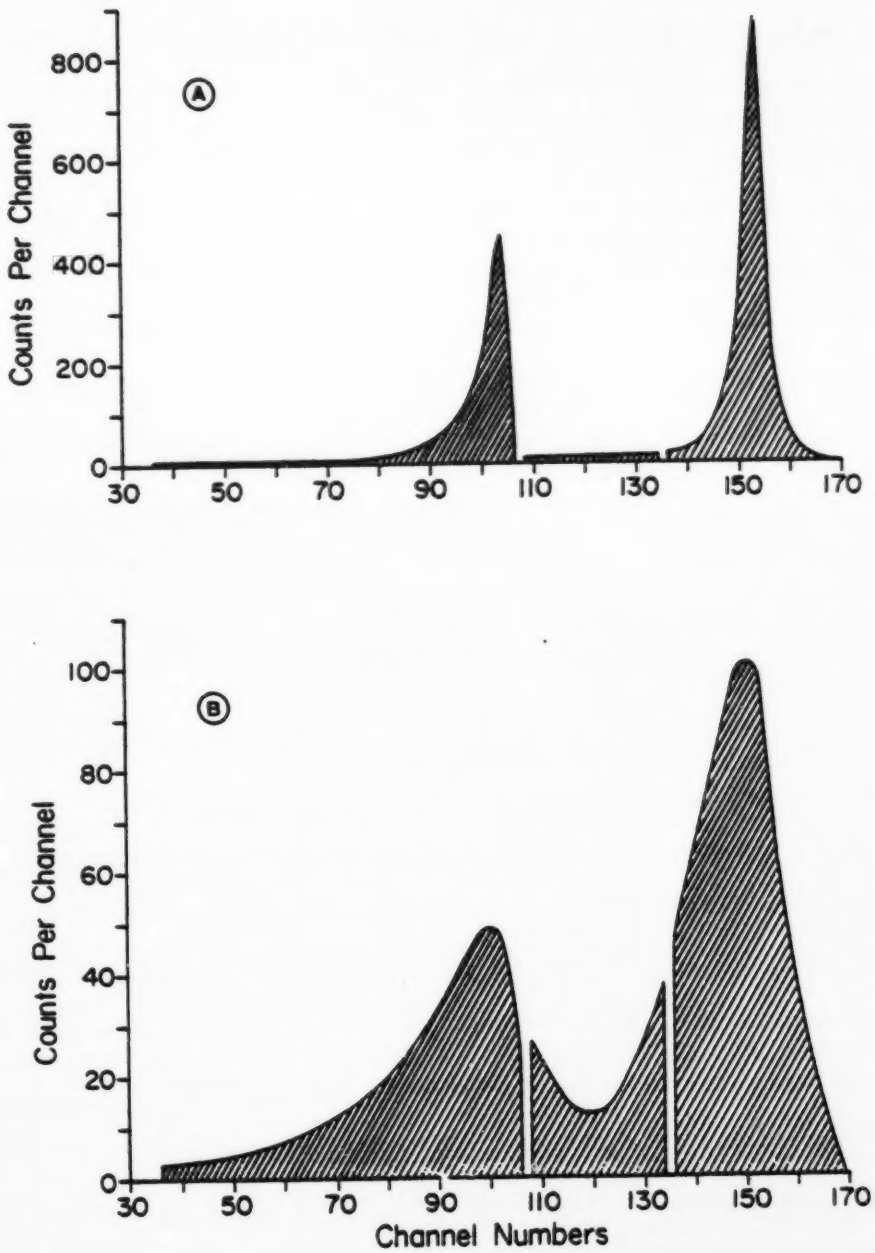


Fig. 5 - Effect of filter loading on peak spreading as it affects energy windows overlapping in instruments using discrimination of alpha-energy as an operating principle.
Note: the regions defined by discriminator voltages are pictured here by making the spectra discontinuous at channels 36, 106 and 135.

spread as the dust load is non-existent. The ThC and ThC' peaks fall neatly within their respective energy windows, with very little ThC' contribution to the middle region. Figure 5b shows the effect of peak spreading from a heavily dust laden filter analyzed under similar conditions as in Figure 5a. The sample was allowed to rest overnight to ensure that radon daughter contributions had vanished. It can be seen that partial energy absorption within the dust load causes the peaks in outside regions to contribute very significantly to counts in the middle region which is ideally reserved for RaC'. This would cause the instrument to erroneously assume the presence of radon daughters in the sample when that particular count was taken. This effect might be minimized by using membrane filters at lower flow rates, especially in very dusty areas. It might prove useful to change the filters more often if this is possible.

CONCLUSIONS

Self-absorption was not found to be a problem when using membrane filters. For glass fiber filters, however, self-absorption was found to vary between 2 and 8%, both in underground mines and within laboratory facilities. It is recommended that, if previous knowledge of the aerosol type is available, corresponding adjustment be made in the sampling flow rate in order to keep self-absorption at a minimum. For example, in the case of Figure 2b, the flow rate should ideally be kept at less than 4 L/min (20 cm/s) as long as this provides sufficient activity for proper counting statistics.

Dust loading of fiber and membrane filters leads to partial and total absorption of alpha energy. This will in most cases affect continuously sampling environmental monitors that operate on the discrimination of alpha-particle energy. This is caused by an overlapping of activity peaks into adjacent energy windows due to a broadening of those peaks. This problem

cannot be eliminated, but can be controlled by using lower flow rates. This has the effect of reducing the effective depth of penetration of nuclides.

ACKNOWLEDGEMENT

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APPENDIX

Derivation of self-absorption formula as outlined in reference 5.

First we assume a filter disc of unit thickness which has been used for sampling. The activity is further assumed to be embedded at a distance X from the front of the filter. At point X we label as D, the actual disintegration rate of radionuclides. Some of the alpha-particles are absorbed by the filter media, hence we label the count rate on the front of the filter as C_1, the count rate on the back of the filter C_2, and the count rate on the front when another filter of unit thickness covers the actual filter as C_3. We also define B as unit absorption coefficient for the filter material. It can be seen that any count rates C_1, C_2 or C_3 are actually the decay rate D minus the portion of counts that is absorbed, hence:

$$C_1 = D - XBD$$
$$C_2 = D - (1 - X)BD \qquad \text{Eq A-1}$$
$$C_3 = D - (1 + X)BD$$

where, X is a distance from the front of the filter where the activity is assumed to be located.

D is the true disintegration rate at depth X.

B is an absorption coefficient.

In order to get D as a function of C_1, C_2 and C_3 alone we perform $2C_1 + C_2 - C_3$ and obtain:

$$D = \frac{2C_1 + C_2 - C_3}{2} \qquad \text{Eq A-2}$$

The self-absorbed fraction is defined as:

$$SA = 1 - \frac{C_1}{D} \qquad \text{Eq A-3}$$

Hence, from Equation A-2 using Equation A-3:

$$SA = \frac{C_2 - C_3}{2C_1 + C_2 - C_3} \qquad \text{Eq A-4}$$